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MATHEMATICAL MODEL DEVELOPMENT FOR VISCOSITY OF TRANSESTERIFIED JATROPHA OIL USING BOX - BEHNKEN METHOD

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ABSTRACT

The objective of this paper was to optimize the viscosity of transesterified Jatropha Curcas biodiesel has gained importance for boosting the fuel industry and improving the production efficiency of biodiesel. A mathematical model for the transesterification of Jatropha Curcas oil was developed at five different process parameters, such as catalyst concentration, reaction time, methanol to oil ratio, stirrer speed and extraction temperature. A three - level - five factorial Box - Behnken was employed to design the experiment. Analysis of variance (ANOVA) of experimental results at 95% confidence level showed that catalyst concentration and reaction time were significant process parameters. The models can be successfully adopted in fuel industry to minimize the viscosity of transesterified Jatropha oil. The prepared Jatropha Curcas biodiesel (JCB) conformed to the ASTM and IS standards specifications.

KEYWORDS: Mathematical Model, Jatropha Oil, Box-Behnken & ANOVA

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Original Article

INTRODUCTION

Recently there has been a colossal increase in the number of automobiles in India. These automobiles release massive pollutants and many other harmful components to the environment, and the resulting air quality deterioration along with it. So the increasing global concern has causes to focus on the oxygenated diesel fuels because of the environmental pollutions from internal combustion engines [1]. Also, world is presently confronted with twin crisis of fossil fuel deflection and environmental degradation. Fossil fuels are limited sources [2].As such, the situation demands for an alternative source of energy that can be used to overcome the forecasted future energy crisis. In addition to this, if the energy source is clean and renewable, it will reduce the environmental issues as well. In this quest for an alternate and renewable energy source, scientists have come up with a variety of options among which vegetable oil as alternative fuels has become a popular option and is gaining the attention of many researchers [3].

When Rudolph Diesel invented the diesel engine, he initially fuelled it with vegetable oil (VGO). However, the usage of VGO in diesel engines suffers from several drawbacks over time, mainly due to their high viscosity. Some of the problems with VGO in diesel engines are for example carbon deposition, injector plugging and piston sticking amongst others [4]. On the other hand, an advantage with biodiesel is comparable properties with diesel and with far lower viscosity than VGO. It can be used blended or sometimes unblended in a diesel engine without modifications [4]. There are four main methods to reduce the viscosity of VGO: blending, micro emulsion, pyrolysis and transesterification [5]. Blending refers to mixing VGO with diesel to achieve an acceptable viscosity. Micro emulsion is a clear fluid consisting of oil and water phases (an alcohol) together with a surfactant. Pyrolysis is decomposition

(cracking) of the oil by means of heating or a catalyst without the presence of oxygen to reduce its viscosity [5, 6]. All three methods reduce the viscosity, but blending and micro emulsion will continue to give the same engine problems as VGO [6]. The product of pyrolysis may be chemically comparable to fossil fuel but the method is very expensive [7]. The last method is transesterification. It is an efficient and commonly used method for reducing the viscosity of VGO and produce biodiesel [4, 7]. It can be done with acidic, alkali or enzymatic catalysts. The acidic and alkali catalysts can be in liquid or solid state, where liquid catalysts are the most common. The general transesterification reaction involves glycerol triesters (oil, triglyceride), changed to alkyl monoesters [4, 7]. Stoichiometric 3 moles of alcohol are needed per mole of triglyceride [4]. The reaction is normally catalyzed with an alkali, but also acidic and enzymatic catalysts are used [5, 6].

The main advantage using biodiesel fuels as 100% methyl (or) ethyl esters of vegetable oil and animal fat (or) biodiesel blends (up to 20% blend to the diesel fuel) are producing less smoke and particulates, having higher cetane numbers and producing lower carbon monoxide and hydrocarbon emissions[8]. The plants such as oil palm, Jatropha, melon, soybean, corn and sugar cane can be produced in large quantities for (use as biomass for use in) biodiesel fuel production. Among these plants Jatropha is of particular importance since it is a non - edible plant and will not be competing for human food as the others [9]. Jatropha oil produced from Jatropha plant could be the best feed stock for biodiesel production [10].

LITERATURE REVIEW

Jatropha oil belongs to the family of vegetable oils that are now promising alternatives in solving energy [11]. Problems due to several advantages; it is renewable, environ – friendly and can be produced easily in rural areas, where there is an acute need for modern forms of energy [12]. However, the properties of these oils are not suitable to be used in engines. They have high viscosity, high flash point, and low calorific value than diesel fuel, thus making them unsuitable to be used in diesel engines. This necessitates the need to go for modification in the oils to make their properties suitable for engine use. Transesterification is the most suitable method to go for modification in oils [13].

Transesterification is the reaction between a lipid and alcohol in the presence of a catalyst to form esters and glycerol. The base catalyst normally used in sodium hydroxide because it is cheaper and safer to handle than potassium hydroxide and the alcohol preferred is methanol which is also cheaper than the other alcohols and gives short chain fuels with desirable properties [14].

Francisca Diana da Silva Araujo et al extracted the oil from the fruit seeds of Jatropha curcas, grown in north eastern Brazil and then characterized, as well as the oxidative stability of Jatropha biodiesel was studied, using Rancimat technique [15]. R.M.Kibuge et al determined that the fuel properties and burning characteristics of sour plum (Ximenia americana L.) seed oil and then which was compared with Jatropha curcas seed oil when unblended and blended with kerosene [16]. Prerna Goyal et al employed that the five – level – four factorial Central Composite Design (CCD) using Response Surface Methodology (RSM) to optimize the process variables for minimizing free fatty acid (FFA) and maximizing the Jatropha curcas biodiesel (%) yield [13].

K.Mu’azu et al developed the mathematical model for the esterification reaction of Jatropha curcas seed oil, in terms of catalyst concentration, methanol to oil ratio and reaction time [17]. Also they developed the mathematical model for the transesterification of Jatropha curcas seed oil by factorial analysis of design of experiment. The factors studied were methanol to oil molar ratio (6-10), catalyst concentration (4 – 8 %), reaction time (1-2 hours) and stirrer speed (100 – 700

rpm) [18]. O.O. Ogunleye et al studied the solvent extraction of oil from Jatropha seed to investigate the effect of process parameters such as temperature, time and solvent composition on the yield and quality of the extract. This also led to the formation and solution of nonlinear programming model for maximizing oil yield subject to biodiesel feedstock requirements [11].

A.Okullo et al carried out a transesterification of Jatropha oil with methanol in a well mixed reactor at different agitation speed (600 – 900 rpm) and temperatures (35 – 65^{o} C) using sodium hydroxide catalyst [14]. Sepidar Sayyar et al optimized that extraction of Jatropha oil from seeds using organic solvent based on the amount of extracted oil. The kinetics of extraction was also investigated and its parameters were determined based on a second order model [19].

LIU Yingying et al investigated that biodiesel produced from crude Jatropha Curcas L. Oil with trace sulphuric acid catalyst (0.02% - 0.08% oil) at 135 -184^{o} C. Both esterification and transesterification can be well carried out simultaneously. Factors affecting the process were investigated, which included the reaction temperature, reaction time, the molar ratio of alcohol to oil, catalyst amount, water content, free fatty acid (FFA) and fatty acid methyl ester (FAME) content [20].

The objective of this paper was to optimize the viscosity of transesterified Jatropha Curcas (JCB) biodiesel by using Response surface methodology (RSM). RSM is a collection of statistical and experimental methods for planning, modelling, evaluating and analyzing variables for a wanted response.

METHODOLOGY

Jatropha curcas seed oil was obtained from National Research Institute for Chemical Technology (NARICT), Zaria, Nigeria. The Jatropha Curcas seeds were of analytical grade and 99% pure and had a moisture content of 6.2% and oil content of 40% [21].The seeds were constantly washed to take off dirt and other impurities. Afterwards it was dried in oven at 40^{o}C until it reached constant moisture content. These seeds were crushed to get particle size ranging from 0.5 mm to 0.75 mm. Then these seeds were taken and centrifuged to separate the solid fraction from the solution. The extracts were heated and evaporated using rotary evaporator apparatus to obtain solvent-free oils. Raw Jatropha curcas oil was filtered to remove all insoluble impurities and foreign particles followed by heating at 100^{o}C for 20 min to remove all the moisture.

PROCEDURE

The transesterification reaction was conducted in a 1-litre reactor-equipped with a reflux condenser, temperature indicator and sampling port, shown in figure 1. This process is conducted using methanol as solvent and CaO (Calcium Oxide) as base catalyst. In the transesterification reaction, the glycerol ester is converted into the methyl ester. The methyl ester layer was separated, washed with water, heated to remove moisture and dried. In the present study, the experiments for transesterification of Jatropha curcas oil has been designed using Response surface methodology (RSM) for the production of Jatropha Curcas biodiesel (JCB). The viscosity of the transesterified Jatropha oil obtained from this process.

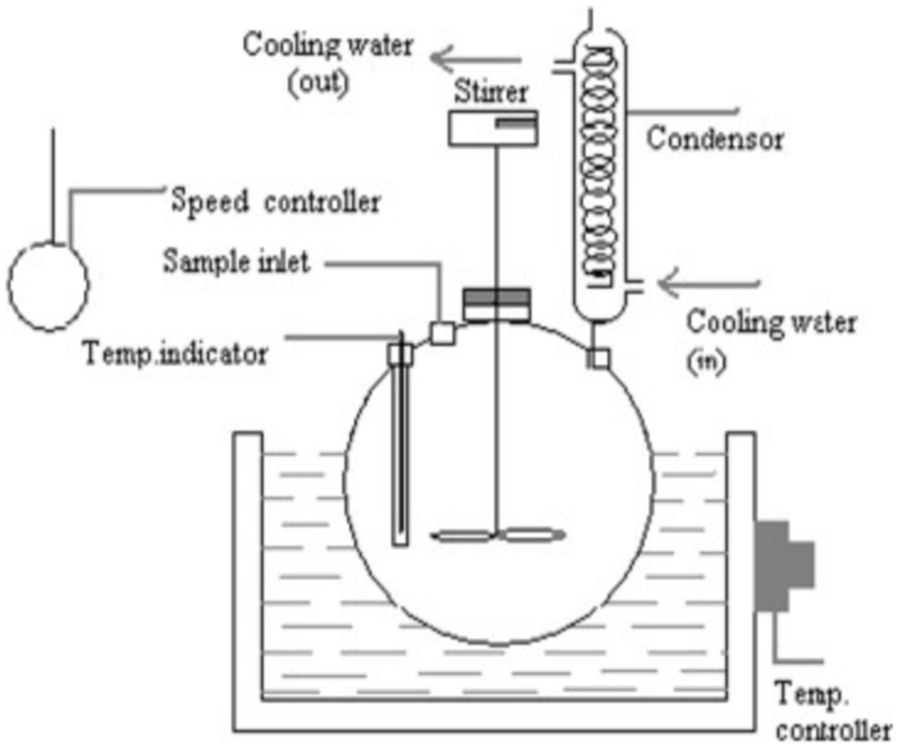

Figure 1: Transesterification Setup

DESIGN OF EXPERIMENT

There are two approaches used in the design of experiments, namely Central composite method and Box-Behnken design. The statistical predictability of Box-Behnken design at 95 % confidence level comparable with some extent with that of central composite design at some extreme conditions, thus indicates the advantage of Box-Behnken design. So, a three-level-five-factor Box-Behnken was applied for carryout the optimization studies to reduce the viscosity of Jatropha oil in the transesterification process. A total of 46 experiments were conducted separately for getting the experimental response of viscosity of JCB. The process parameters selected for the transesterification process were catalyst concentration, methanol to oil molar ratio, extraction temperature, reaction time and stirrer speed. The description of the process parameters and output responses for the transesterification of Jatropha oil is given in Table 1.

Table 1: Description of Process Parameters and Output Responses for the Transesterification of Jatropha Oil

FACTORS	UNITS/MEASURES	DESCRIPTION	REFERENCES
Input Process Parameters	Catalyst concentration (% w/w)	It is the percentage of catalyst (CaO) contained in unit weight of Jatropha Curcas oil.	Muazu.K et al (2012) [18], K. Mu'azu et al (2013) [17], O. O. Ogunleye et al (2012) [11], Prerna Goyal et al (2012) [13].
	Methanol to oil molar ratio (w/w)	It is the ratio of unit weight of methanol to unit weight of Jatropha Curcas oil.	Muazu.K et al (2012) [18], K. Mu'azu et al (2013) [17], Prerna Goyal et al (2012) [13].

	Reaction time (minutes)	It is the time taken to complete the transesterification process.	Muazu.K et al (2012) [18], K. Mu'azu et al (2013) [17], O. O. Ogunleye et al (2012) [11], Prerna Goyal et al (2012) [13].
	Stirrer speed (RPM)	It is the speed of the stirrer in revolutions per minutes.	Muazu.K et al (2012) [18].
	Extraction temperature (^oC)	It is the temperature at which the transesterification process taking place.	O. O. Ogunleye et al (2012) [11], Prerna Goyal et al (2012) [13].
Output Response	Viscosity of Transesterified Jatropha oil (m^2/s)	It is the kinematic viscosity of Jatropha Curcas oil after the transesterification process.	Muazu.K et al (2012) [18], K. Mu'azu et al (2013) [17], Prerna Goyal et al (2012) [13].

The coded levels of the process parameters used for the transesterification of Jatropha oil are given in Table 2.

Table 2: The Coded Levels of Process Parameters Used for the Transesterification of Jatropha Oil

Factors	Symbols	Levels		
		-1	0	1
Catalyst concentration(% w/w)	A	1	4	7
Methanol to oil molar ratio (w/w)	B	7	9	11
Reaction time (minutes)	C	60	90	120
Stirrer speed (RPM)	D	100	400	700
Extraction temperature (^oC)	E	54	57	60

DEVELOPMENT OF MATHEMATICAL MODEL

The MINITAB14 software was used for the regression and graphical analysis of the data. From the design experiment for transesterification process the maximum values of Jatropha curcas biodiesel yield and minimum values of viscosity of transesterified Jatropha oil were taken as the responses. The predicted response for viscosity of transesterified Jatropha oil was obtained by the response surface regression using the following polynomial equation (1).

Viscosity of Transesterified Jatropha oil, γ = $ß_o + ß_1 A + ß_2 B + ß_3 C + ß_4 D + ß_5 E + ß_6 A^2 + ß_7 B^2 + ß_8 C^2 + ß_9 D^2 + ß_{10} E^2 + ß_{11} AB + ß_{12} AC + ß_{13} AD + ß_{14} AE + ß_{15} BC + ß_{16} BD + ß_{17} BE + ß_{18} CD + ß_{19} CE + ß_{20} DE$ (1)

Where γ is the predicted response of viscosity of transesterified Jatropha oil, $ß_o$ is model constant and $ß_1$, $ß_2$, $ß_3$, $ß_4$, $ß_5$, $ß_6$, $ß_7$, $ß_8$, $ß_9$, $ß_{10}$, $ß_{11}$, $ß_{12}$, $ß_{13}$, $ß_{14}$, $ß_{15}$, $ß_{16}$, $ß_{17}$, $ß_{18}$, $ß_{19}$ and $ß_{20}$ are coefficients associated with variables A, B, C, D and E and their respective interactions calculated using linear regression method. The model terms were either accepted or rejected based on the P-values with 95% confidence level [22]. Statistical analysis of the model was carried out to evaluate the Analysis of variance (ANOVA).

VALIDATION OF THE MODEL

The experimental data were substituted in the model equation at various conditions of catalyst concentration,

methanol/oil, reaction time stirrer speed and extraction temperature in order to determine the accuracy of the model developed and the corresponding viscosity of transesterified Jatropha was obtained as shown in the following in the Table 3.

Table 3: Matrix Design of the Transestrification Reaction using Actual Factors

A: Catalyst Concentration (%)	B: Methanol /Oil Ratio	C: Time (min)	D: Stirrer Speed (RPM)	E:Extraction Temperature (^oC)	Viscosity of Transesterified Jatropha Oil in m^2/s (γ)	
					Experimental Response	Predicted Response
4	9	90	400	57	6.62	6.21
4	9	120	400	60	6.82	6.40
1	9	120	400	57	6.88	6.44
4	9	60	700	57	5.78	5.12
4	11	90	700	57	6.89	5.49
7	9	90	700	57	6.22	5.26
4	9	60	400	54	6.18	5.65
4	11	90	400	60	6.72	6.07
7	7	90	400	57	6.94	6.40
4	9	90	700	54	5.48	4.42
4	7	90	400	60	6.87	6.29
4	9	90	400	57	6.64	6.21
7	9	120	400	57	6.91	6.58
1	11	90	400	57	6.59	6.24
4	9	120	700	57	7.05	6.81
1	9	90	400	60	6.89	6.12
1	9	90	100	57	7.41	7.26
4	9	90	100	60	7.21	7.24
7	9	90	100	57	7.37	7.08
4	9	90	400	57	6.69	6.22
4	9	90	400	57	6.29	6.21
1	9	90	400	54	6.68	6.15
4	7	60	400	57	6.54	6.05
1	7	90	400	57	6.71	6.24
4	9	120	400	54	7.39	6.60
4	9	120	100	57	7.52	7.27
4	11	90	400	54	6.61	6.20
4	7	120	400	57	7.18	6.48
7	9	90	400	60	6.70	6.05
4	9	90	700	60	5.89	5.00
4	7	90	100	57	7.45	7.67
4	11	90	100	57	6.98	6.87
4	7	90	700	57	6.11	6.04
7	11	90	400	57	6.47	6.05
4	11	60	400	57	5.68	5.45
4	11	120	400	57	7.17	6.73
4	9	90	400	57	6.73	6.21
4	9	60	100	57	7.19	6.96
4	9	60	400	60	5.75	5.65
4	9	90	400	57	7.08	6.21
4	7	90	400	54	6.69	6.34
1	9	90	700	57	5.89	5.09
1	9	60	400	57	6.18	5.73
7	9	60	400	54	5.90	5.56
4	9	90	100	54	7.12	7.09
7	9	90	400	54	6.68	6.20

RESULTS AND DISCUSSIONS

The 3D response surface profile and its contour of the optimal production of bio-diesel based on the equation above are shown in Figure 2. It clearly shows that the catalyst concentration around 4.0% would most likely maximal production of viscosity of bio-diesel. The viscosity increased when the catalyst concentration was outside this range. Since the methanol and triglyceride in the Jatropha curcas are immiscible, addition of catalyst can facilitate the transesterification reaction, and rapidly decrease the viscosity of JCB. However, when the catalyst concentration was too high, soap could be quickly formed which made the separation of glycerol from bio-diesel more difficult, this increase the viscosity of JCB. In contrast, inadequate usage of catalyst could result in an incomplete reaction and a higher the viscosity.

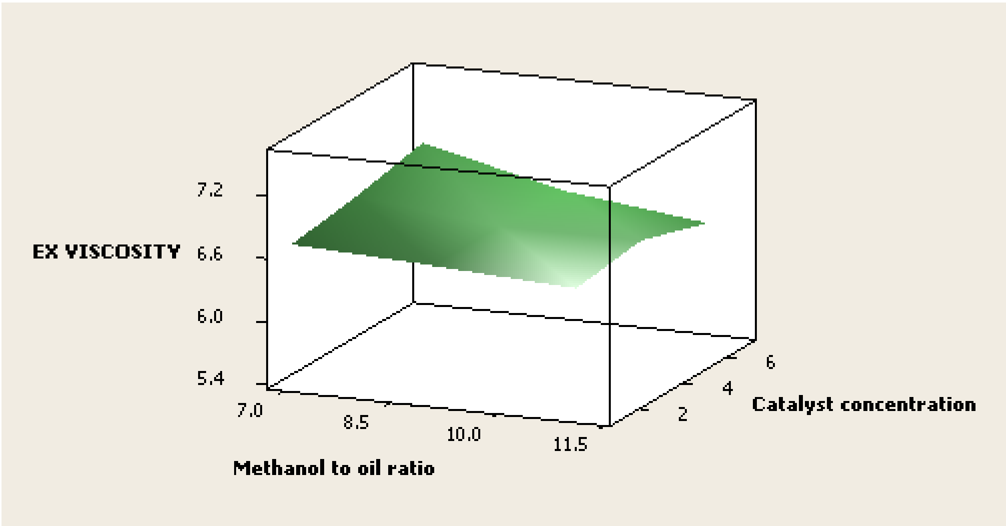

Figure 2: Response Surface Plots Describing the Effects of Process Parameters Interactions on Experimental Viscosity of Transesterified Jatropha Oil

The RSM shown in Figure 2 shows the optimal value of the methanol/oil molar ratio for the viscosity, and that too high or too low values of the methanol/oil ratio have negative effects. This can be explained by the fact that the transesterification is an equilibrium reaction in which excess alcohol will, on the one hand, drive the reaction to the right, decreasing viscosity; but on the other hand will help increase the solubility of glycerol resulting in the reaction driven to the left, thus increasing the viscosity. Too low a methanol/oil molar ratio also led to an incomplete reaction. Therefore, both catalyst concentration and methanol/oil molar ratio exhibited optimal values. The RSM demonstrated that the optimal conditions for catalyst concentration and methanol/oil molar ratio were about 4%.

However, once the catalyst concentration was greater than its centre point value, the reverse trend was observed. A similar pattern was observed when increasing the methanol/oil molar ratio. This could be due to the fact that the positive coefficient for A and B played a major role when the catalyst concentration and methanol/oil molar ratio were at lower levels, while at higher levels, the interaction term AB and quadratic terms A2 and B2 had a more significant negative effect, leading to a increase the viscosity. This was consistent with physical considerations; that since the methanol and triglyceride in the sunflower oil are immiscible; addition of catalyst can facilitate the transesterification reaction, and rapidly decrease the viscosity. However, when the catalyst concentration became too high, soap could be quickly formed which made the separation of glycerol from bio-diesel more difficult, thus reducing the yield. Similarly, the increase of the amount of methanol will, on one hand, drive the reaction to the right since the transesterification reaction is an equilibrium

process; but on the other hand excess methanol will help increase the solubility of glycerol resulting in the reaction being driven to the left, increasing the viscosity of Transesterified Jatropha oil.

The experimental and predicted values for Jatropha Curcas biodiesel yield response at the design points and all the five variables in uncoded form are given in Table 3. ANOVA results of the model showed that the associated Probability (P) value for the model was lower than 0.001, thus, implying the significance of the model. The value of regression coefficient R^2 for the model was 0.93, indicating the good fitness of the model.

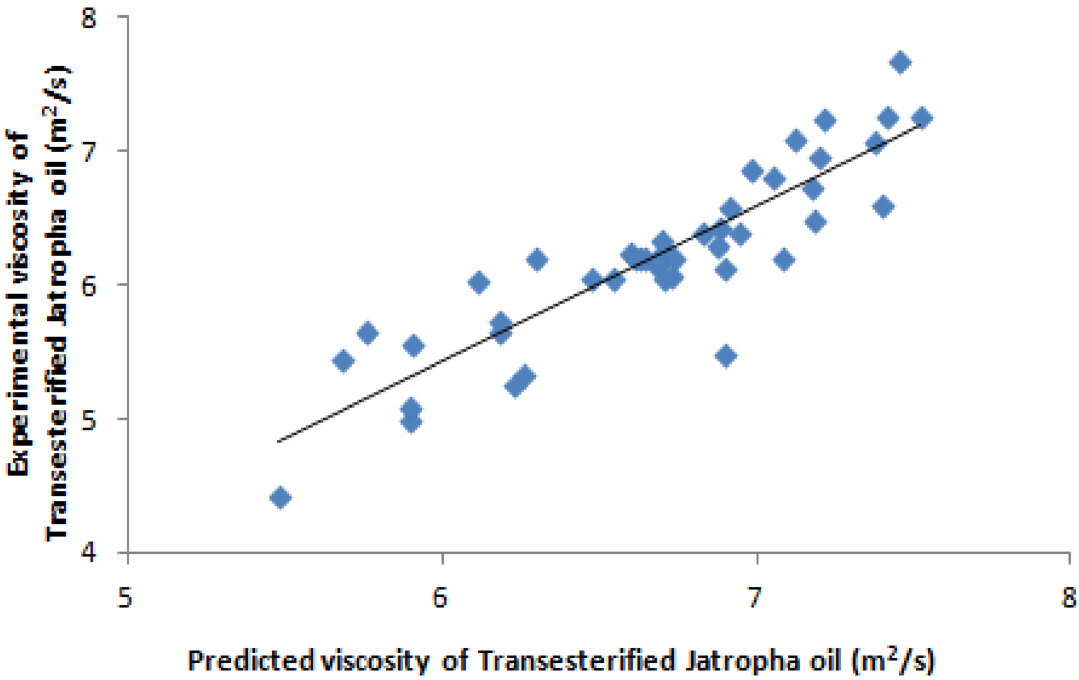

Figure 3: Experimental Versus Predicted Viscosities of Transesterified Jatropha Oil

The figure 3 shows that the predicted values are quite close to the experimental values, thus, validating the credibility of the model developed for establishing a correlation between the process variables and viscosity of transesterified Jatropha oil. The ANOVA for viscosity of transesterified Jatropha oil is given table 4. It shows that catalyst concentration and reaction time were most contributing factors for the viscosity of transesterified Jatropha oil.

Table 4: Anova Table for Viscosity of Transesterified Jatropha Curcas Oil

Dependent Outcome	Independent Measure that have Significant Partial Regression Coefficients	Std. Error	F	P
Viscosity of Transesterified Jatropha oil	Catalyst concentration (A)	2908.9	26.05	0.000
	Reaction time (C)	4637.0	8.33	0.001

CONCLUSIONS

The experimental results were showed that the optimal condition as methanol: oil molar ratio, 9:1, and catalyst concentration, 4%, at reaction time 90 min, and temperature 54°C. Generally more time give higher yield and lower viscosity within the levels chosen for this study. The maximum effect on Jatropha appears to be at 90 minutes. Maximum effect means the level of the variable where the variable it is compared against can be altered the most while still achieving the best response. Reduced time for the viscosity can be achieved with increased temperature. This optimized condition

was validated with actual viscosity of transesterified Jatropha oil of 95%. ANOVA results reveal that reaction time and catalyst concentration are the significant factors that affect viscosity. Temperature is of absolute importance for the transesterification and should be as high as possible. The temperature might even be higher than the boiling point of methanol with methods for sealing the reactor, cooling the vapour and back flow.

The Box-Behnken method has effectively been used in determining the linear and quadratic relationship between the viscosity and the five factors considered for the study. It is also realised that the reaction time and catalyst amounts affect the viscosity greatly. The models can be successfully adopted in fuel industry to minimize the viscosity of transesterified Jatropha oil. The prepared Jatropha Curcas biodiesel (JCB) conformed to the ASTM and IS standards specifications.

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